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Thermal Disorder in Adsorbed Cl on Si(100)

by

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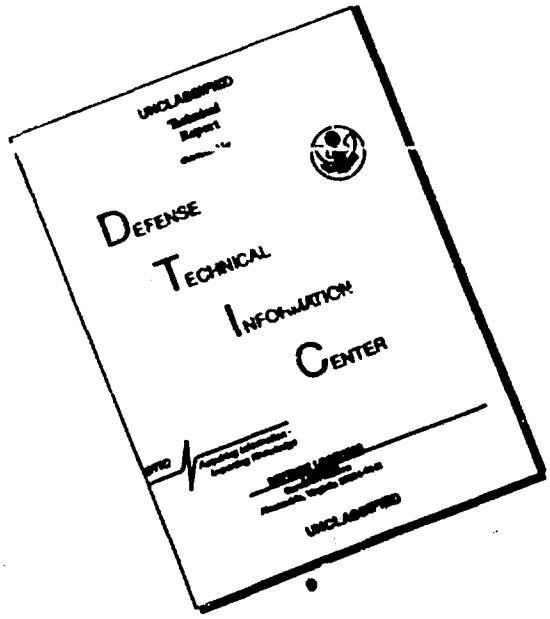

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Abstract

Thermal broadening of Cl^+ ESDIAD patterns, related to the vibrational motion of the adsorbate, has been investigated by subtraction of patterns measured at different temperatures. In the case of Cl on Si(100), the observed difference pattern between 130 K and 305 K indicates that azimuthally isotropic motion of Cl occurs about the most probable Si-Cl bond direction. Equal distributions from the in-plane and out-of-plane bending motions are observed, correlating with the ~200 cm^{-1} frequencies associated with these motions.

The investigation of adsorbate vibrational motions with components parallel to the surface is important for achieving a deeper understanding of two types of surface processes. Surface diffusion and surface reaction processes can be envisioned as involving large amplitude vibrations of adsorbates with components parallel to the surface. The adsorbate vibrations may be considered as movement along the early portion of the reaction coordinate for either surface diffusion, or surface reaction with neighbor adsorbates [1]. Measurements of the vibrational amplitudes of adsorbates on single crystal surfaces can thus provide detailed insights into the shape of the potential energy surface governing adsorbate diffusion and possibly surface reaction. It might be expected that covalent solids with highly directional surface and internal bond directions would exhibit large anisotropies in adsorbate vibrational amplitudes. Here, we report the first observations of the vibrational amplitudes of an adsorbate on a single crystal surface of a covalent solid.

ESDIAD (electron stimulated desorption ion angular distribution) [2] has been used to observe vibrational motions of Cl adsorbed on Si(100) based on thermally-induced pattern broadening. The ESDIAD technique has been employed previously in numerous cases to image adsorbate bonding directions on single crystal surfaces [3]. During the ESD process, the adsorbate interacts with the incoming electrons and undergoes a Franck-Condon type electronic excitation. As a result, a molecule or ion, produced by excitation to a repulsive potential surface, escapes from the substrate by transferring potential energy into kinetic energy [4,5]. In a theoretical analysis of the Franck-Condon ionization efficiency of an adsorbed species, Clinton has shown that ESDIAD patterns image the vibrational wave functions of the surface species [6]. Therefore, it would be expected that peak maxima observed in ESDIAD patterns indeed correspond to the average bonding orientation of the adsorbate, provided the desorbing particle's final state

interaction with the surface is azimuthally isotropic [6]. In addition, Clinton suggested that the intensity spread in the observed ESDIAD pattern is due to vibrational bending motions of the adsorbate [6]. Thus, the ESDIAD patterns obtained at a non-zero temperature would be expected to have contributions from excited vibrational levels. From the nth vibrational level, the number of ions desorbing at a certain angle, α (relative to the most probable bond direction), is obtained from the probability densities $|\Psi_n(\alpha)|^2$, where $\Psi_n(\alpha)$ are the vibrational wave functions. The angular distribution, $\langle N(\alpha)\rangle$, for a non-degenerate normal mode is then given approximately by the thermal average over all these contributions:

$$\langle N(\alpha)\rangle \approx (1/Q) \sum_{n=0}^{\infty} |\Psi_n(\alpha)|^2 \exp[-(E_n-E_0)/kT], \qquad (1)$$

where Q is the vibrational partition function and $(E_n-E_0) = nh\nu$, the energy above the zero-point energy of the nth vibrational level with the vibrational frequency, ν.

The dynamical behavior of surface species has been recently investigated using the ESDIAD method [7]. The summation of millions of individual ESD events in an ESDIAD pattern, resulting from adsorbed species on identical bonding sites of a single crystal surface, yields a direct picture of the statistical distribution of adsorbate positions with respect to the equilibrium position at that temperature, and hence a view of the vibrational excursions of the adsorbate in various directions along the surface. This is because within the Franck-Condon approximation, the electronic excitation time is short compared to the time for one vibration of an adsorbate. Using a digitized data acquisition method, ESDIAD has been successful in observing rotational as well as vibrational motions of adsorbates

on single crystal metal surfaces [7]. In this report, we refine the use of digital ESDIAD to observe adsorbate dynamics by using a pattern subtraction method [8]. Upon subtraction of two high quality ESDIAD patterns obtained at different temperatures, the difference pattern displays the regional gains or losses of ion emission intensity, attributable to the thermal disorder of the adsorbate on its site.

The choice of the Si(100) surface in this study was made because of its technological importance. In addition, one also expects highly directional adsorbate bonding geometry from a covalent solid. The Si(100) surface has been well studied and is known for its (2x1) reconstruction [9]. The reconstruction is a result of the dimerization of surface atoms. Consequently, the Si(100)-(2x1) surface consists of parallel rows of Si dimers, and the surface dangling bond density is decreased from 2 dangling bonds per surface atom on the idealized unreconstructed (1x1) surface to 1 dangling bond per atom on the (2x1) surface. Adjacent terraces separated by monoatomic steps on Si(100) exhibit (2x1) and (1x2) domains [10]. Chemisorption of Cl atoms on these highly directional dangling bonds thus provides an ideal system for the study of surface bonding as well as the dynamical behavior of the Si-Cl bond using ESDIAD.

The ultrahigh vacuum (UHV) system (with a base pressure of 3×10^{-11} Torr) and the Si(100) crystal preparation have been described previously [11]. The UHV system is equipped with a CMA Auger electron spectrometer (AES), an argon ion sputtering gun, a digital LEED/ESDIAD apparatus, a shielded quadrupole mass spectrometer (QMS) for line-of-sight thermal desorption studies, and a second QMS associated with an electron gun for ion mass analysis in ESD. The Si(100) crystal (orientation accuracy = 1°; 15 x 15 x 1.5 mm; p-type; B-doped; 10 Ω-cm) is resistively heated (120 K - 1200 K) by a Honeywell programmable temperature controller used to drive a feedback circuit to control the heating power to the crystal [12]. Dosing of molecular species was done by using a multicapillary-

collimated doser with a known angular distribution and from which the molecular flux was calibrated [13]. The identity of the ionic species (Cl^+) produced from ESD on the Cl-covered surface was determined by the auxiliary QMS with the internal ionizer turned off. The mass spectrometry intensity ratio of m/e = 35 to m/e =37 is found to be 3:1, consistent with the natural abundance of isotropic Cl. All ESDIAD measurements were made with an electron energy of 120 eV. Typical electron beam currents were 5 nA, avoiding significant beam damage during the measurement of ESDIAD patterns. For all the data presented here, a crystal bias potential (+ 10.0 V) was used to compress the Cl^+ trajectories. The result of the ion trajectories compression does not affect the qualitative interpretation presented here. No electrical heating current flows through the crystal during the measurement. The digital ESDIAD data have been azimuthally smoothed using a procedure described previously [14]. The azimuthal smoothing method assumes, based on the two-fold symmetry of the Si(100)-(2x1) + (1x2) surface, that ion intensities originating from adsorbates at a given polar angle and in azimuthal directions 180° apart should be equal.

Bonding structures of Cl on Si(100) have been investigated by ESDIAD previously [15,16]. The terminal-bonded Cl, produced by adsorption of Cl_2 at 673 K, is bonded with a Si-Cl bond oriented 25° ± 4° from the surface normal in a vertical plane parallel to the Si-Si dimer bond direction ($<01\bar{1}>$ on the (2x1) terrace. Figure 1(a) shows the Cl^+-ESDIAD pattern obtained after annealing a monolayer of Cl at 673 K for 60 s; the pattern was measured at 130 K. The off-normal Cl^+-ESD beams observed indicate the Si-Cl bonds are tilted away from the surface normal, and the four-beam pattern is a consequence of the superposition of two pairs of two-fold azimuthally symmetric patterns originating from the two perpendicular domains on the Si(100) surface [10,15,16].

Thermal broadening of the angular distribution of Cl^+-ESD beams is

observed on the same surface by measurements at higher temperatures. Figure 1(b) shows the broadened Cl^+-ESDIAD pattern measured at 305 K. Within the temperature range 130 K - 305 K, no surface chemical reactions are observed [15,16], and the thermal broadening effect is a reversible process. In order to see the details of the broadening process, the difference of pattern (b) and pattern (a) is shown in Figure 1(c). The total yields represented in the patterns shown in Figures 1(a) and 1(b) are of the order of 10^7 Cl^+ counts. The difference pattern shown in Figure 1(c) has a total positive contribution of ~ 10^6 Cl^+ counts matched by a negative contribution of the same magnitude . The difference pattern (Figure 1(c)) can be divided into two regions: (1) negative-intensity regions observed at four positions which correspond to the peak positions before subtraction; (2) positive-intensity regions observed in the central area and also at positions away from the <011> and <011> axes. The difference intensities distributed around the outside edges are near zero which can serve as a "datum level" for viewing the difference pattern.

Figure 2 shows sections A and B made across the ESDIAD difference pattern of Figure 1(c). Section A displays the enhanced Cl^+ intensity contribution along vertical planes at 45° angles to the <011> and <01$\bar{1}$> directions (45° from the Si_2 dimer bond directions). Section B displays the gain and loss of Cl^+ intensity in the two Si_2 dimer bond directions along the vertical planes containing the Si_2 bonds. The loss in Cl^+ intensity in the most probable Si-Cl bond direction is readily observed as well as the gain in Cl^+ intensity in the normal direction to the surface and along the 45° directions.

Considering the normal modes for Cl on Si(100) (see Figure 3), the motions responsible for the thermal broadening will correspond to the two bending modes of the Si-Cl bond on the surface. The frequency for the Si-Cl bending modes is in the range 150 - 250 cm^{-1}, based on vibrational spectroscopic studies of gaseous

chlorosilane compounds [17], and is ~ 220 cm^{-1} according to HREELS studies of $SiCl_4$ adsorbed on Si(100) [18]. The vibrational frequency of the Si-Cl stretching mode has been determined by high resolution electron energy loss spectroscopy (HREELS) on Si(100), and is in the range ~550 - 600 cm^{-1} depending on coverage [16]. We neglect the Si-Cl stretching vibration since it is of minor importance for our present analysis, and it is energetically too high to make a significant contribution in the temperature range studied here. On the other hand, the out-of-plane bending motion will cause the displacement of Cl perpendicular to the vertical <011> and <01$\bar{1}$> planes, leading to azimuthal broadening as observed at angles away from the <011> and <01$\bar{1}$> directions. The in-plane bending motion will cause the displacement of Cl parallel to the Si_2 dimer bond due to the change of the Cl-Si-Si bond angle. For both in-plane and out-of-plane motions, contributions to difference features in the ESDIAD patterns are produced by superposition of intensity from pairs of geometrically distinct Si-Cl surface species. The four observed negative-intensity regions are expected from the depopulation of the vibrational ground states of the two bending modes.

In examining the vibrational contributions to the observed Cl^+-ESDIAD pattern, we will use the approximate frequency of 200 cm^{-1} estimated for the Si-Cl bending modes [17,18] and the bending force constant, k_b, of $SiCl_4$ (0.628 mdyn Å rad^{-2}) [19] for the following analysis. Using a harmonic-oscillator approximation, partitioning into the nth vibrational level can be calculated as follows:

$$P(n) = [\exp(-nh\nu/kT)]\,[1 - \exp(-h\nu/kT)], \qquad (2)$$

where P(n) is the thermal population in the nth level. In addition, the simplest estimate of the angular vibrational amplitude is given by the classical

approximation for torsional simple harmonic motion,

$$E_n = (n + 1/2)\, h\nu \approx 1/2\, k_b\, \alpha_n^2, \qquad (3)$$

where α_n is the classical limit of the angular vibrational amplitude for the oscillator in the nth vibrational level. Results for P(n) and α_n (expressed in degrees) based on Eqns. 2 and 3 are tabulated in Table 1. The population change, $\Delta P(n)$, in the first excited state between the two temperatures (130 K and 305 K) is 14.1 %. There are also significant contributions from higher excited vibrational levels which add another ~ 14 % to the overall depletion of the ground state. The increase of population in the excited levels is coupled with an increase in vibrational amplitudes. For the first two excited levels with the most significant increases in population between the two temperatures, the classical amplitudes are 7.9° and 10.2° for the first and second excited levels, respectively compared to only 4.6° for the ground state. The 28 % decrease in population of the ground-state vibrational level corresponds to the negative-intensity regions of the difference ESDIAD pattern. This analysis suggests that thermal broadening of the Cl^+ ESDIAD patterns is primarily due to the Si-Cl bending motions.

We have modeled the vibrational behavior of the Si-Cl bond, demonstrating good fits to Gaussian distributions around the most probable bond directions for two experimental patterns at different temperatures (Figures 1(a) and (b)). The following parameters were employed in the modeling: (1) an increase of full width at half maximum (FWHM) of the Gaussian distributions by 20%, based on the measured beam widths of the Cl^+ ESDIAD patterns;(2) an increase in the integrated areas of Gaussian distributions by 10%, corresponding to the experimentally measured value of the change in Cl^+ ion yields, and (3) a small decrease in polar angle as the temperature increases. This shift observed at 305 K

is measured to be ~5% of the measured polar angle at 130 K (see Figures 1(a) and (b)) and its origin is not explored here. The resulting difference pattern is shown in Figure 4. It may be seen that the modeled difference pattern in Figure 4 closely matches the experimental ESDIAD difference pattern of Figure 1(c). The cross-section views in <011> and <010> directions of the model difference pattern, shown as dashed lines in Figure 2, are also in good agreement with the experimental measurements. Anisotropic broadening of Gaussian peaks was also modeled. For changes of FWHM anisotropically larger than 8%, significant differences in the modeled patterns are observed. Thus, we conclude that isotropic broadening of the Si-Cl bond directions best describes the thermal disorder experienced by the chemisorbed Cl atoms.

Although the above analysis, restricted to the vibrational properties of the adsorbed state (initial state), provides a good qualitative correlation between the observed ESDIAD pattern broadening and the vibrational behavior of the adsorbate, a full interpretation requires considerations of the behavior of the departing Cl^+ ion (final state). Two prominent final state effects are: (1) path-dependent neutralization probabilities of the desorbing ions (a short-range effect), and (2) deflection of the outgoing ions by their own electrostatic image charge in the substrate (mainly a long-range effect). These two effects in ESDIAD have been discussed in detail elsewhere [14,20-23]. Other effects like initial momentum broadening of the wavepacket (representing the desorbing particle) [24,25] and lattice vibrations will also contribute to the observed widths of the ESDIAD pattern.

In summary, using the digital ESDIAD technique coupled with a pattern subtraction method, we have been able to spatially image the vibrational motion of Cl adsorbed on Si(100). While it might be expected that large anisotropies in vibrational motion would be observed for an adsorbate on a structurally-corrugated

covalent-bonded solid surface, we find instead that the vibrational motion is essentially azimuthally isotropic. This is in accordance with the similarity of the frequency for in-plane and out-of-plane bending modes in chlorosilane molecules.

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Figure Captions

Figure 1. (a) Cl^+-ESDIAD pattern taken at 130 K; (b) Cl^+-ESDIAD pattern taken at 305 K; (c) pattern obtained by subtraction of (a) from (b). The figures on the left are perspective plots; they are generated by mapping the ion intensity into the z direction. The scale of pattern (c) in the z direction is 1/5 of that of patterns (a) and (b). The figures on the right are contour plots. Each contour line in each individual plot represents an increment of 1/7 of the plot's maximum. The width of the contour is ± 5% of the median value of that contour. Four symmetric regions of Cl^+ intensity loss are seen, along with intensity gain in a cross-shaped region containing a central peak.

Figure 2. Cross-section views of the ESDIAD difference pattern shown in Figure 1(c). Dashed lines depict a model involving isotropic broadening of the four Cl^+ ESDIAD beams.

Figure 3. Schematic of the normal modes of Cl on Si(100).

Figure 4. The model ESDIAD difference pattern with the assumption of isotropic thermal broadening of the Si-Cl bond direction.

Table 1. Calculations of vibrational characteristics of the Si-Cl bending modes with $\nu = 200\ cm^{-1}$ and $k_b = 0.628$ mdyn Å rad^{-2}. α_n is classical amplitude; P(n) is Boltzmann state population.

n	α_n	P(n) (%) 130 K	P(n) (%) 305 K	$\Delta P(n)^a$
0	4.6	89.1	61.1	- 28.0
1	7.9	9.7	23.8	+ 14.1
2	10.2	1.1	9.2	+ 8.1
3	12.1	0.1	3.6	+ 3.5
4	13.7	---	1.4	+ 1.4

a. $\Delta P(n) = P(n, 305\ K) - P(n, 130\ K)$

ESDIAD Patterns of Cl on Si(100)

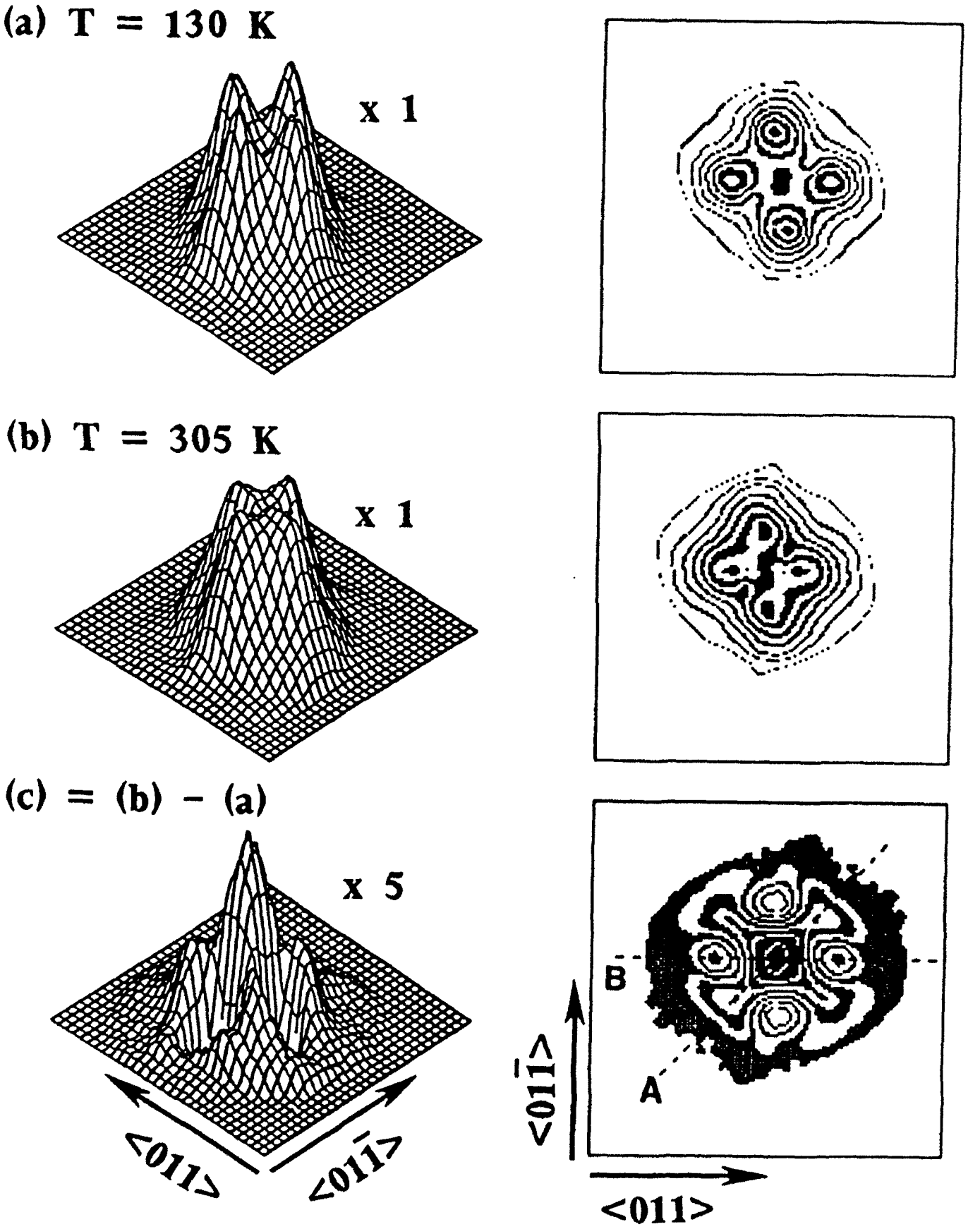

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Figure 1

Cross Sections of ESDIAD Difference Pattern for Two Particular Directions

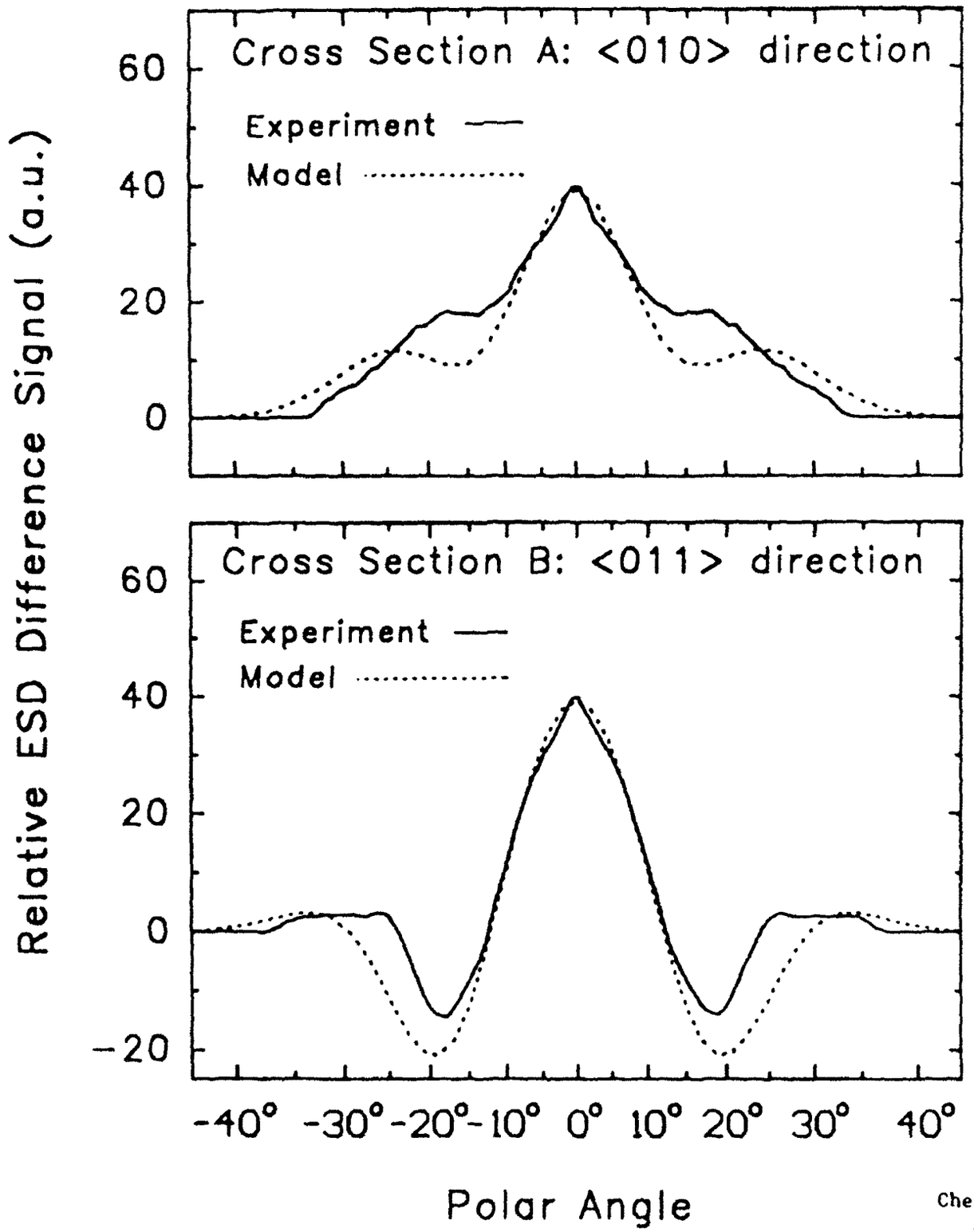

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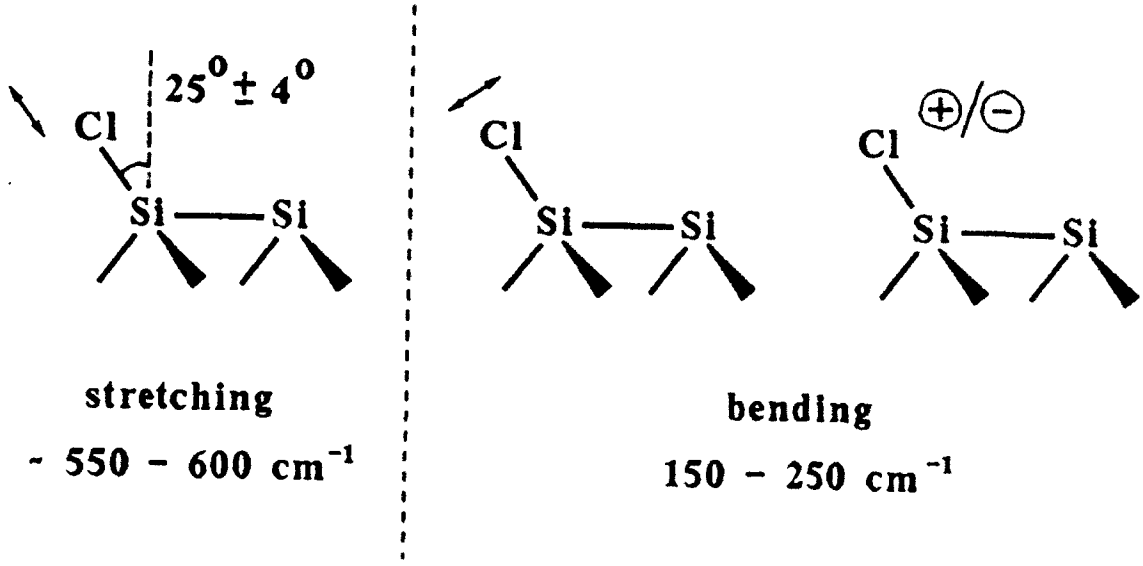
Figure 2

Schematic of the Normal Modes of Cl on Si(100)



stretching

~ 550 - 600 cm^{-1}

bending

150 - 250 cm^{-1}

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Figure 3

Modeled ESDIAD Difference Pattern of Cl on Si(100)

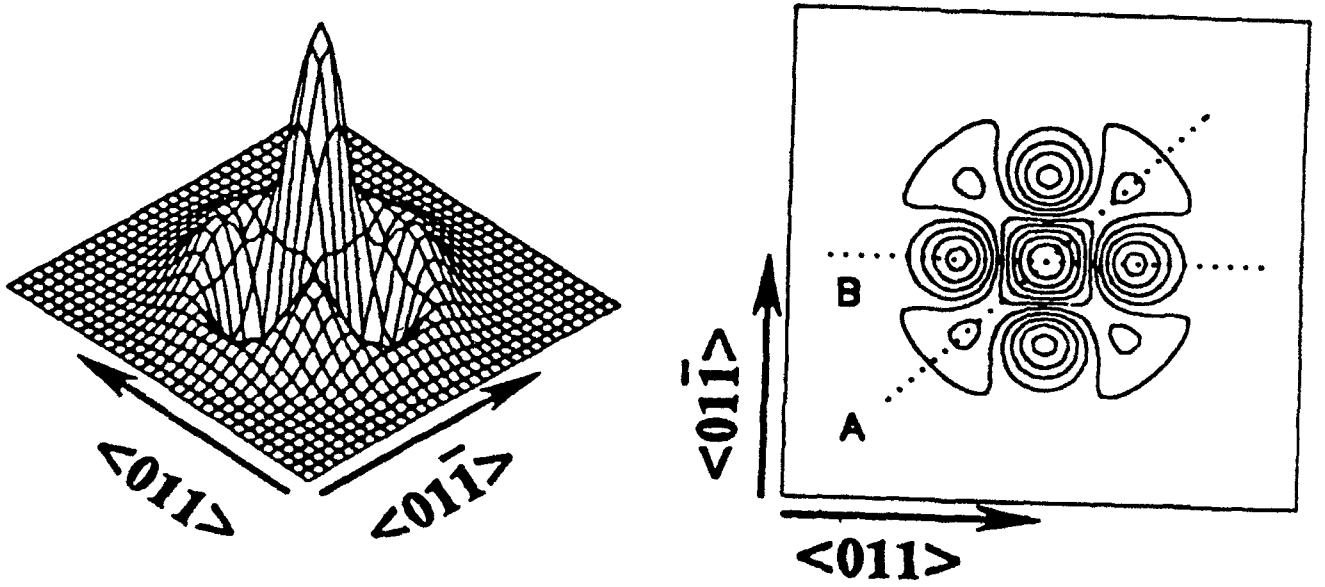

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Figure 4

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